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PALLADIUM TETRAAZANULENES: THE SYNTHESIS AND STRUCTURE OF PALL--ETC(U)
OCT 78 M TSUTSUI, R L BOBSEIN, G CASH

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Palladium Tetraazaannulenes: The Synthesis and

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Tetramethyl-Dibenzo(b,i)(1,4,8,11)Tetraaza(14)Annulene.

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by

M. Tsutsui, R. L. Bobsein, G. Cash, and R. Pettersen

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PALLADIUM TETRAAZAANULENES: THE SYNTHESIS AND STRUCTURE
OF PALLADIUM 5,7,12,14 - TETRAMETHYL-DIBENZO[b,1][1,4,8,11]TETRAAZA[14]ANNULENE

M. Tsutsui, R. L. Bobsein, G. Cash and R. Pettersen

Department of Chemistry
Texas A&M University
College Station, Texas 77843

Abstract:

The first palladium dibenzotetraaza[14]annulenes have been prepared from the reaction of the free base ligand with bis(benzonitrile)palladous chloride. Analytical data are consistent with the proposed structures. Palladium 5,7,12,14-tetramethyl-dibenzo[b,1][1,4,8,11]tetraaza[14]annulene was characterized by single-crystal x-ray diffraction.

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INTRODUCTION

Palladium coordination complexes prefer a square planar arrangement and are known to be among the most stable.^{1,2} Ligands such as porphyrins, phthalocyanines, and tetraazaannulenes prefer to coordinate with a metal ion in a square planar fashion. The fact that palladium porphyrins are very resistive to chemical demetallation illustrates the stability of palladium square planar complexes.³ In contrast to square planar complexes, 5,7,12,14-tetramethyl-dibenzo[b,1][1,4,8,11]tetraaza[14]annulene coordinates with metal ions in a square pyramidal arrangement.⁴ Now we wish to report a new coordination geometry in the palladium complex of this ligand.

Dibenzotetraaza[14]annulene has been complexed with a variety of metals;⁵⁻¹³ some of these complexes exhibit unusual geometries.¹⁴⁻¹⁶ Metal tetraazaannulenes have been known to possess chemical and thermal stability as well as high catalytic activity, particularly as electrocatalysts.¹⁷⁻¹⁹ A cobalt tetraaza[14]annulene has been used as the photoactive substituent in a potential solar energy storage system.²⁰

To our knowledge, no one has reported a palladium complex of tetraaza[14]annulene. Although these macrocyclic ligands generally influence the physical and chemical properties of the metal,²¹ an x-ray diffraction analysis of this first palladium tetramethyl-tetraazaannulene shows that palladium metal insertion has the effect of flattening a hitherto saddle-shaped ligand into a pseudo-planar configuration.^{4,22,23}

EXPERIMENTAL SECTION

Bis(benzonitrile)palladous chloride²⁴ and 1,8-dihydro-5,7,12,14-tetramethyl-dibenzo[b,1][1,4,8,11]tetraaza[14]annulene (H_2TMTAA)⁸ were mixed in refluxing benzene. The title compound, PdTMTAA, was obtained from the resulting

precipitate. PdTMTAA crystallized as orange platelets in hexane. PdTMTAA decomposes at 150°C to a yellow-brown compound, which melts at 260°C. Analysis²⁵ calculated for $\text{Pd}(\text{C}_{22}\text{H}_{22}\text{N}_4)$: C, 58.87; H, 4.94; N, 12.48; Pd, 23.90. Found: C, 59.18; H, 5.12; N, 12.20; Pd, 23.51. The visible spectrum of PdTMTAA in dichloromethane has two peaks at 415nm. ($\log \epsilon$ 4.5) and at 490nm. ($\log \epsilon$ 4.2). The infrared spectrum of PdTMTAA in the solid phase (KBr) shows peaks at 1475, 1395, 1070, 820 and 790 cm^{-1} . The mass spectrum is consistent with the structure of PdTMTAA.²⁶ The parent ions around 448 m/e appear in an isotopic pattern typical of naturally occurring palladium.

The palladium complex of 1,8-dihydrodibenzo[b,i][1,4,8,11]tetraaza[14]annulene (H_2TAA)⁵ has also been prepared. The above complex, PdTAA, was synthesized in a manner similar to the previously described PdTMTAA synthesis. PdTAA was purified by sublimation. Analysis²⁵ calculated for $\text{Pd}(\text{C}_{18}\text{H}_{14}\text{N}_4)$: C, 55.05; H, 3.59; N, 14.26; Pd, 27.09. Found: C, 54.95; H, 3.59; N, 14.01; Pd, 27.56. The visible spectrum of PdTAA is a typical metal insertion spectrum with peaks at 363, 402, 424, 455, and 487 nm. in chloroform. The spectrum is very similar to that of NiTAA⁵ indicating that PdTAA is a planar molecule.²⁷

CRYSTAL EXAMINATION AND DATA COLLECTION

All crystals of PdTMTAA examined were extremely thin plates, the large faces of which were parallelograms. The one selected for x-ray work measured 0.23x0.22x0.008 mm and was mounted with the fiber axis approximately parallel to the longest edges.

Data were collected by Molecular Structure Corporation, College Station, Texas. Details are presented in Table I. The following equations were used in reducing the data; L_p is the Lorentz-polarization factor, S is the scan rate, C is the total integrated peak count, B is the total background count, and R is the ratio of scan time to background counting time: $I = S(C - RB)$, $\sigma(I) = [S^2(C + R^2B) + 0.0025I^2]^{1/2}$, $|F_o| = (I/L_p)^{1/2}$, $\sigma(F_o) = \sigma(I)/(2|F_o|L_p)$. Periodic checks

of three standard reflections revealed no deterioration during data collection. On the basis of 4 scans, absorption corrections were deemed unnecessary. The linear absorption coefficient, μ , is 10.65 cm^{-1} .

SOLUTION AND REFINEMENT OF THE STRUCTURE

The structure was solved using the crystallographic multi-purpose program SHELX (by George Sheldrick); thermal ellipsoid plots were generated by ORTEP (thermal ellipsoid plotting program by C. K. Johnson). These computations were performed on the Amdahl 470V/6 computer at Texas A&M University.

Systematic absences ($h0\ell$, $h + \ell \neq 2n$ and $0k0$, $k \neq 2n$) unambiguously identified the space group as $P2_1/n$. Since $Z = 2$, it was necessary that the palladium atoms lie on centers of symmetry. A Patterson map confirmed this. A difference Fourier revealed all the carbon and nitrogen atoms. Refinement of these, followed by a difference map, revealed all the hydrogen atoms also. When all the atoms had been refined to convergence (hydrogen atoms isotropic, others anisotropic), a difference map revealed no peak larger than $0.4 \text{ e}/\text{\AA}^3$. The final unweighted R , defined as $\Sigma |F_o - |F_c|| / \Sigma F_o$, was 0.036; the weighted R_w , $(\Sigma w |F_o - |F_c||^2 / \Sigma w F_o^2)^{1/2}$, was 0.046. The weight, w , is given by $w = k / (\sigma^2(F_o) + g F_o^2)$; k was redetermined after each cycle and g was refined at the same time by fitting $(F_o - F_c)^2$ to $(\sigma^2(F_o) + g F_o^2) / k$. The final values of k and g were 0.7731 and 8.93×10^{-4} , respectively. For the nonhydrogen atoms, the largest shift in a positional parameter on the last cycle was 0.35σ . Scattering factors used in these calculations were taken from ref. 28. Cromer and Liberman's values²⁹ for the real and imaginary components of anomalous dispersion due to palladium were used.

DESCRIPTION AND DISCUSSION OF THE STRUCTURE OF PdTMTAA

The final atomic coordinates are listed in Tables II and III. A listing

of interatomic distances and bond angles are provided in Tables IV and V for PdTMTAA. Figure 1 is an ORTEP plot of PdTMTAA. This view of the structure is very similar to other published structures^{4,7,21} in the general geometry of the ligand. The four Pd-N distances average 1.996Å which is longer than the corresponding metal nitrogen distances of 1.870Å^o observed for NiTAA and 1.901Å^o observed for CoTMTAAI. This increase is expected because of the larger ionic radius of palladium. A comparison of M-N distances in various metal-lomacrocycles and crystal ionic radii of the metal atom is summarized in Table VI. The data in Table VI show that as the size of the metal increases, one expects a concomitant displacement of the metal from the N₄ plane or some kind of ligand distortion.²¹ Possibilities for relieving ligand strain introduced by metal insertion include some combination of (1) an increase in interatomic distances on the 14-membered ring, (2) an increase in some of the interior angles of the ring and (3) a deformation of the ring carbon atoms from the N₄ plane. The palladium atom in PdTMTAA does not deviate from the N₄ plane as required by symmetry. The atoms of the 14-membered ring are displaced from the N₄ plane by a maximum of .467Å^o, as illustrated in Figure 2. There are appreciable distortions in the five-membered chelate rings and in the benzenoid rings but this pseudo-planar geometry contrasts with the very non-planar geometry of other published TMTAA structures.^{11,21} In the latter compounds, the methyl groups interact sterically with the benzenoid rings so as to warp or distort the ligand, thus relieving steric strain. It has been suggested that the size of the metal ion directly influences the degree of distortion.¹¹ In the case of PdTMTAA, the opposite effect is observed. The palladium complexation has flattened the normally saddle-shaped ligand, H₂TMTAA.

The total angular change in the ten interior and four exterior angles as defined in Figure 3 is an important measure of the degree of expansion of

the ring (see Table VII). The sum of the values of the ten interior and four exterior angles for H_2TMTAA , $FeTMTAA$, $CoTMTAAI \cdot CHCl_3$, and $FeTMTAACl \cdot CH_3CN$ is smaller than the corresponding sum in $PdTMTAA$ by 6.4° , 16.8° , 26.2° , and 19.4° , respectively. Only $MnTMTAA \cdot N(Et)_3$ shows a total angular expansion (2.2°) compared with $PdTMTAA$. This similar total angular increase in the manganese compound does not permit the smaller manganese ion to sit in the N_4 plane. In this case the manganese ion also has a large axial ligand which may prevent the metal ion from sitting in the plane.

Since interatomic bond extensions are unlikely to occur because they are energetically unfavorable, it appears that ring expansion through increases in some interior bond angles provides most of the additional space required for the palladium ion to fit in. This factor probably influences the ability of the metal and the macrocycle to achieve the pseudo-planar configuration.

A comparison of some dihedral angles is also presented in Table VII. The dihedral angles as defined in Figure 2 are a measure of the distortion of the macrocyclic ligand. As can be seen from Table VII, TMTAA complexes of manganese, iron, and cobalt, as well as the free ligand, have a very large amount of distortion yielding the characteristic saddle shape. In contrast, $PdTMTAA$ has, in general, about half the distortion. The dihedral angle between planes 6 and 7 is larger for $PdTMTAA$ than H_2TMTAA , $MnTMTAA \cdot N(Et)_3$, and $FeTMTAACl \cdot CH_3CN$ and mainly indicates that the methyl group is displaced slightly out of the ligand plane. In fact, the nonbonding interatomic distance between the methyl group and the nearest carbon atom of the benzenoid ring for $PdTMTAA$ is 2.925\AA , about $.2\text{\AA}$ shorter than the corresponding distance in the manganese compound. Thus, in $PdTMTAA$ there exists comparatively little warping of the macrocyclic ligand and the methyl groups tilt slightly from the plane of the macrocycle as they get closer to the benzenoid rings. Table VII

also presents a comparison of some selected bond lengths. The bond lengths of the PdTMTAA molecule are similar to those of some of the previously characterized TMTAA's. The average C-N distance in the six-membered ring in PdTMTAA shows more double bond character. The average C-C distance in the six-membered chelate rings is comparable in all seven compounds. The average C-N distance on the five-membered chelate ring shows more single bond character in all seven compounds.

Thus, it has been shown that palladium coordination to the TMTAA ligand yields a unique geometry. The strain generated by this pseudo-planar geometry is not alleviated by increased bond lengths or warping. Only the slight displacement of the methyl groups out of the plane and the angular increase may account for this planarity. Such an effect might be explained in terms of an especially strong palladium coordination sphere. In order to maximize such interactions, the tetramethyl-tetraazaannulene ring distorts from its usually stable saddle shape. First row transition metal complexes of the TMTAA ligand exhibit this saddle shape. Perhaps extended interactions by the larger palladium d orbitals force the ligand into its pseudo-planar configuration.

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Supplementary Material Available: A listing of observed and calculated structure factors (5 pages).

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Table I. Data Collection and Refinement Details for $\text{Pd}(\text{C}_{22}\text{H}_{22}\text{N}_4)$ (PdTMTAA)

Diffractionmeter	Enraf-Nonius CAD-4 ^a
Monochromator	Graphite Crystal
Radiation	Mo K α (λ 0.71069Å)
Take off angle	2.8°
Scan speed	4° to 20° / min
Scan width	1°
Standards	3
2 θ Limits of data	50°

^a Data taken by Molecular Structure Corporation, College Station, Texas 77840.

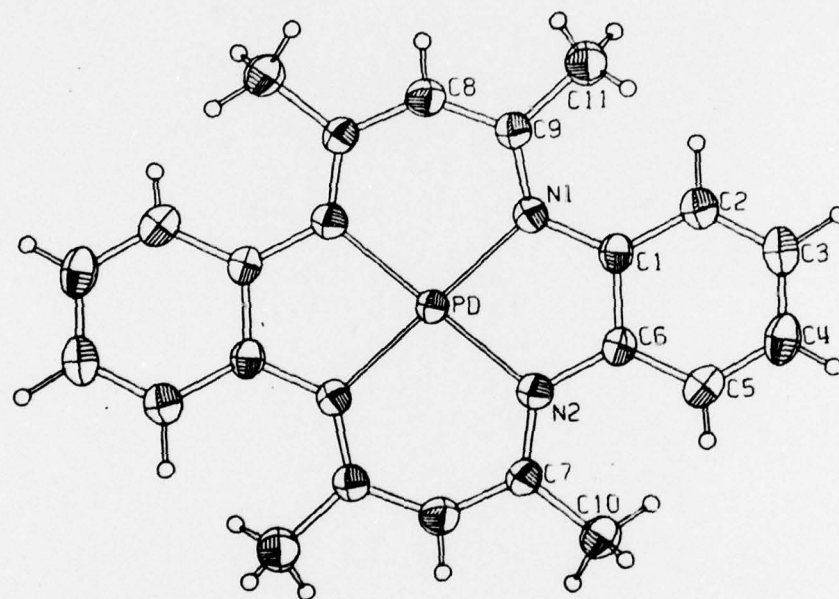


Fig. 1
M. Tsutsui

Figure 1. ORTEP of PdTMTAA

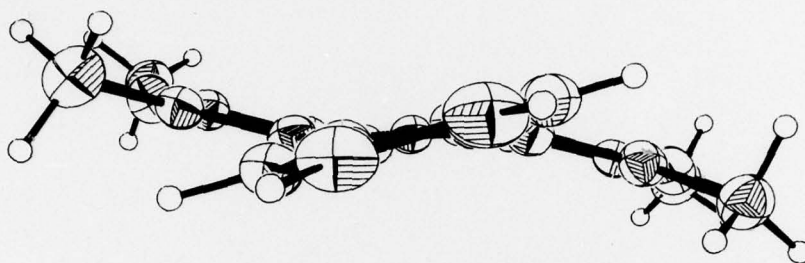


Figure 2. A side view of $\text{Pd}(\text{C}_{22}\text{H}_{22}\text{N}_4)$ illustrating deviations from the containing Pd and N's. (Space group symmetry requires that these five atoms be in the same plane.)

Table II. Final Positional and Thermal Parameters of Nonhydrogen Atoms for $[\text{Pd}(\text{C}_{22}\text{H}_{22}\text{N}_4)]$

ATOM	X	Y	Z	U11	U22	U33	U12	U13	U23
Pd	0.0(0)	0.0(0)	0.0(0)	0.0234(3)	0.0250(3)	0.0251(3)	0.0002(4)	0.0085(2)	0.0005(4)
N1	-0.0126(8)	0.0563(3)	0.2260(7)	0.0221(25)	0.0293(27)	0.0268(27)	0.0035(21)	0.0053(21)	-0.0026(22)
N2	0.2027(8)	0.0768(3)	-0.0287(7)	0.0277(27)	0.0316(27)	0.0269(28)	0.0021(23)	0.0092(22)	0.0015(23)
C1	0.1270(10)	0.1144(3)	0.2556(9)	0.0253(32)	0.0293(32)	0.0311(34)	-0.0020(26)	0.0024(26)	-0.0026(27)
C2	0.1852(11)	0.1578(3)	0.4181(10)	0.0350(36)	0.0333(35)	0.0325(36)	0.0041(30)	0.0073(29)	0.0001(30)
C3	0.3008(12)	0.2194(4)	0.4213(11)	0.0422(42)	0.0315(35)	0.0438(43)	0.0000(32)	0.0017(34)	-0.0123(33)
C4	0.3619(13)	0.2403(4)	0.2642(11)	0.0452(44)	0.0247(36)	0.0555(51)	-0.0017(33)	0.0073(36)	0.0011(34)
C5	0.3228(11)	0.1972(4)	0.1107(11)	0.0283(35)	0.0322(36)	0.0442(43)	0.0011(28)	0.0112(32)	0.0145(32)
C6	0.2227(9)	0.1302(3)	0.1093(9)	0.0223(30)	0.0237(30)	0.0336(35)	0.0031(25)	0.0058(25)	0.0021(26)
C7	0.3366(10)	0.0675(3)	-0.1382(9)	0.0297(34)	0.0287(33)	0.0310(34)	-0.0026(27)	0.0128(28)	0.0040(28)
C8	0.3124(11)	0.0116(4)	-0.2650(9)	0.0382(34)	0.0368(43)	0.0330(32)	-0.0024(33)	0.0200(27)	0.0045(32)
C9	0.1714(10)	-0.0462(3)	-0.3088(9)	0.0290(35)	0.0307(34)	0.0256(33)	0.0025(28)	0.0063(27)	0.0032(26)
C10	0.5315(11)	0.1123(4)	-0.1240(11)	0.0328(38)	0.0396(41)	0.0427(43)	-0.0049(33)	0.0150(33)	0.0013(35)
C11	0.2190(13)	-0.0972(4)	-0.4495(12)	0.0405(44)	0.0430(45)	0.0474(48)	-0.0027(36)	0.0220(36)	-0.0104(37)

The anisotropic parameters (\AA^2) are used in the expression $\exp(-[2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^{*}b^{*} + 2U_{13}hla^{*}c^{*} + 2U_{23}k\ell b^{*}c^{*})])$.

TABLE III. Final Positional and Thermal Parameters of Hydrogen Atoms for $[\text{Pd}(\text{C}_{22}\text{H}_{22}\text{N}_4)]$

ATOM	X	Y	Z	U
H2	0.1357(105)	0.1423(34)	0.5310(91)	0.0242(174)
H3	0.3422(109)	0.2473(33)	0.5459(98)	0.0387(188)
H4	0.4361(108)	0.2784(36)	0.2651(94)	0.0311(186)
H5	0.3634(117)	0.2116(34)	0.0171(100)	0.0343(189)
H8	0.4211(106)	0.0103(31)	-0.3209(87)	0.0278(162)
H10A	0.4855(111)	0.1547(36)	-0.1947(100)	0.0380(203)
H10B	0.5854(97)	0.1317(34)	-0.0002(92)	0.0251(165)
H10C	0.6282(118)	0.0820(36)	-0.1737(99)	0.0383(198)
H11A	0.3605(122)	-0.0892(35)	-0.4464(94)	0.0306(184)
H11B	0.1355(165)	-0.0896(46)	-0.5826(141)	0.0968(311)
H11C	0.2096(124)	-0.1430(42)	-0.4123(111)	0.0287(239)

Table IV. Interatomic Distances in $[\text{Pd}(\text{C}_{22}\text{H}_{22}\text{N}_4)]$ (PdTMTAA), Å

Pd - N1	1.999 (5)
Pd - N2	1.993 (5)
Pd - C1	2.839 (6)
Pd - C6	2.840 (6)
C1 - N1	1.395 (8)
C6 - N2	1.413 (8)
C9 - N1	1.340 (8)
C7 - N2	1.342 (8)
C1 - C2	1.424 (9)
C1 - C6	1.411 (9)
C2 - C3	1.370 (10)
C3 - C4	1.380 (11)
C4 - C5	1.366 (10)
C5 - C6	1.408 (9)
C7 - C8	1.388 (9)
C7 - C10	1.498 (9)
C8 - C9	1.400 (9)
C9 - C11	1.502 (9)

Table V. Bond Angles for PdTMTAA, deg.

N2 - Pd - N1	83.0 (2)
C1 - N1 - Pd	112.3 (4)
C6 - N2 - Pd	111.9 (4)
C7 - N2 - Pd	121.2 (4)
C7 - N2 - C6	125.1 (5)
C2 - C1 - N1	126.8 (6)
C6 - C1 - N1	115.8 (5)
C6 - C1 - C2	117.4 (6)
C3 - C2 - C1	121.3 (7)
C4 - C3 - C2	119.7 (7)
C5 - C4 - C3	120.4 (7)
C6 - C5 - C4	121.2 (7)
C1 - C6 - N2	115.7 (5)
C5 - C6 - N2	126.2 (6)
C5 - C6 - C1	118.1 (6)
C8 - C7 - N2	121.8 (6)
C10 - C7 - N2	123.3 (6)
C10 - C7 - C8	114.7 (6)
C9 - C8 - C7	133.9 (6)
C11 - C9 - C8	114.4 (6)

TABLE VI. Average N - M Distance for Various Macrocycles^a and Empirical Crystal Ionic Radii of Metal Atoms^b

	M - N Distance	Ionic Radii
PdTMTAA	1.996 ^o Å	0.86 ^o Å
FeTMTAA	1.918 ^o Å	0.76 ^o Å
H ₂ TMTAA (N - H)	.85 ^o Å [1.902 ^o Å (N - Ct)]	
PdTPP	2.009 ^o Å	0.86 ^o Å
FeTPP	1.972 ^o Å	0.76 ^o Å
FeTPPCl	2.05 ^o Å	0.64 ^o Å
FePc	1.926 ^o Å	0.76 ^o Å
FeOctaaza[14]annulene	1.836 ^o Å	0.76 ^o Å
FeTMTAACl	2.002 ^o Å	0.64 ^o Å
CoTMTAAI	1.901 ^o Å	0.63 ^o Å
MnTMTAA · N(Et) ₃	2.118 ^o Å	0.80 ^o Å
NiTAA	1.870 ^o Å	0.72 ^o Å
NiC ₂₀ H ₁₈ N ₄ (NiDMTAA)	1.85 ^o Å	0.72 ^o Å

^aRefs. 7, 11, 21, 30

^bRef. 31

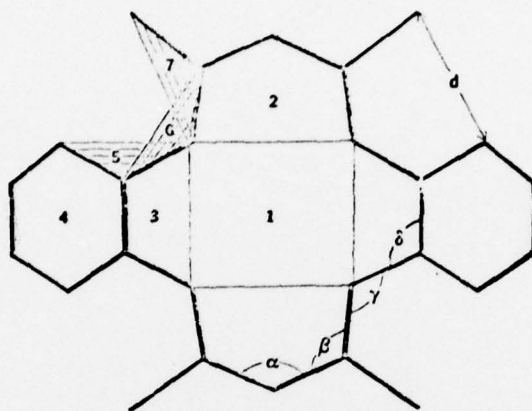


Figure 3. Definition of bond angles.

From M. C. Weiss et al., J. Am Chem. Soc., 98, 8021 (1976).

TABLE VII. Average Bond Angles, deg.^a; Comparison of Average Dihedral Angles, deg.^a; and Selected Average Interatomic Distances, Å

	PdMTAA	H ₂ TMTAA ^a	FeTMTAA ^b	MnTMTAA·N(Et) ₃ ^c	FeTMTAACl·CH ₃ CN ^c	CoTMTAAI·CHCl ₃ ^c	NiTAA ^d
1-2 ^a	α 133.9	127.3	129.9	131.2	129.6	130.2	124.1
1-3	β 122.1	120.1	121.6	123.1	121.5	121.4	124.7
1-4	λ 125.3	127.4	125.3	126.3	125.0	123.8	120.2
5-6	δ 115.7	117.3	114.0	115.6	113.9	113.2	113.6
6-7	14.7	34.3	24.5	35.2	32.9	21.3	-
	1.4	20.1	17.5	12.4	16.3	16.2	-
	13.0	24.6	23.7	20.5	21.8	21.7	-
	25.0	45.6	31.5	51.3	43.5	28.5	-
	5.2	3.6	9.27	1.6	3.2	12.1	-
Av. M-N distance	1.996	-	1.918	2.118	2.002	1.901	1.870
Distance of M from N ₄ donor plane	0.000	~ 0.1(H's)	.114	0.730	0.600	0.234	0.000
Ct-N distance	1.996	1.902	-	1.988	1.910	1.893	1.870
Av. C-N distance six-membered chelate ring	1.341	1.330	1.342	1.327	1.331	1.344	1.327
Av. C-C distance six-membered chelate ring	1.393	1.387	1.391	1.406	1.404	1.382	1.398
Av. C-N distance five-membered chelate ring	1.404	1.404	1.418	1.403	1.422	1.411	1.418

TABLE VII. (continued)

d ^a	2.925	3.098	3.016	3.126	3.054	2.949	-
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^aSee Figure 3.

^bRef. 4.

^cRef. 11.

^dRef 21.

SUPPLEMENTARY MATERIAL AVAILABLE

OBSERVED AND CALCULATED STRUCTURE FACTORS

OBSERVED AND CALCULATED STRUCTURE FACTORS														PAGE 1
M	K	L	10FQ	10FC	M	K	L	10FQ	10FC	M	K	L	10FQ	10FC
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6	0	0	524	542	4	9	0	69	33	5	0	1	385	372
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3	1	0	508	514	1	10	0	90	-82	-4	1	1	444	466
4	1	0	132	-119	2	10	0	413	410	-3	1	1	224	-230
5	1	0	989	503	4	10	0	391	370	-2	1	1	944	940
7	1	0	213	214	6	10	0	186	184	0	1	1	1068	1064
1	2	0	1037	1169	1	11	0	379	394	1	1	1	344	-332
1	2	0	746	-658	3	11	0	368	387	2	1	1	595	616
2	2	0	793	741	4	11	0	161	-158	4	1	1	596	560
3	2	0	156	162	5	11	0	231	233	6	1	1	345	348
4	2	0	579	584	0	12	0	405	422	-7	2	1	141	139
5	2	0	157	147	1	12	0	159	155	-6	2	1	102	93
6	2	0	359	358	2	12	0	349	347	-5	2	1	527	525
1	3	0	890	926	4	12	0	284	286	-3	2	1	667	654
2	3	0	347	-368	6	12	0	217	214	-2	2	1	294	276
3	3	0	831	799	1	13	0	373	387	0	2	1	521	-510
5	3	0	384	397	3	13	0	452	449	1	2	1	1556	1508
7	3	0	271	281	5	13	0	305	314	2	2	1	133	132
0	4	0	471	475	0	14	0	496	498	3	2	1	551	545
1	4	0	349	354	2	14	0	253	247	5	2	1	270	271
2	4	0	713	713	4	14	0	342	333	6	2	1	134	-129
4	4	0	339	348	1	15	0	383	374	7	2	1	289	299
6	4	0	370	355	3	15	0	409	424	-6	3	1	341	352
1	5	0	497	520	5	15	0	228	212	-5	3	1	156	-146
2	5	0	675	588	0	16	0	451	454	-4	3	1	440	431
3	5	0	988	972	1	16	0	158	-159	-2	3	1	646	630
4	5	0	162	-172	2	16	0	358	369	-1	3	1	124	-125
5	5	0	225	215	4	16	0	243	233	0	3	1	820	825
7	5	0	293	284	1	17	0	308	318	1	3	1	1309	-1314
1	6	0	166	165	2	17	0	74	33	2	3	1	541	546
2	6	0	658	645	3	17	0	294	310	3	3	1	463	444
4	6	0	451	437	0	18	0	368	377	4	3	1	463	450
6	6	0	239	245	2	18	0	352	355	6	3	1	261	269
1	7	0	625	626	4	18	0	220	227	7	3	1	91	-71
3	7	0	213	198	1	19	0	312	320	-7	4	1	234	222
5	7	0	219	223	3	19	0	210	234	-5	4	1	437	407
7	7	0	312	304	0	20	0	293	270	-4	4	1	246	-237
1	8	0	206	214	1	20	0	95	-97	-3	4	1	539	550
3	8	0	666	670	2	20	0	258	264	-2	4	1	104	-108
5	8	0	107	98	1	21	0	216	227	-1	4	1	763	775
7	8	0	534	549	0	22	0	153	168	0	4	1	107	104
2	8	0	364	359	-5	0	1	404	402	1	4	1	1142	1169
5	8	0	40	-97	-3	0	1	813	818	2	4	1	485	-457
6	8	0	129	147	-1	0	1	690	682	3	4	1	521	521
1	9	0	282	293	1	0	1	408	413	5	4	1	387	366

M	K	L	10FO	10FC	M	K	L	10FO	10FC	M	K	L	10FO	10FC
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-3	10	1	113	-118	3	5	1	113	-118	-3	10	1	460	459
-2	10	1	315	288	4	5	1	315	288	-2	10	1	257	247
-1	10	1	186	183	6	5	1	186	183	-1	10	1	597	536
0	10	1	118	-95	7	5	1	118	-95	0	10	1	156	-145
1	10	1	298	279	-7	6	1	298	279	1	10	1	465	459
2	10	1	314	318	-5	6	1	314	318	2	10	1	64	60
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0	13	1	438	439	-3	8	1	438	439	0	13	1	362	356
2	13	1	216	-202	-2	8	1	216	-202	2	13	1	344	341
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-1	14	1	367	342	5	8	1	367	342	-1	14	1	374	383
0	14	1	265	254	-6	9	1	265	254	0	14	1	145	134
1	14	1	391	385	-4	9	1	391	385	1	14	1	472	475
3	14	1	115	106	-3	9	1	115	106	3	14	1	350	365

OBSERVED AND CALCULATED STRUCTURE FACTORS

PAGE 2

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-5 15	1	80	60	0	1	2	308	314	1	6	2	110	-108	2 11	2	332	324	-1 19	2	160	156			
-4 15	1	261	257	1	1	2	445	461	2	6	2	833	842	3 11	2	271	287	0 19	2	74	65			
-2 15	1	250	251	2	1	2	95	-99	4	6	2	157	162	5 11	2	333	346	1 19	2	205	199			
0 15	1	592	598	3	1	2	342	346	6	6	2	255	270	-6 12	2	193	217	3 19	2	184	195			
1 15	1	126	126	5	1	2	325	323	-7	7	2	245	254	-4 12	2	437	449	-2 20	2	191	183			
4 15	1	329	313	-6	2	2	211	206	-5	7	2	486	481	-2 12	2	323	343	0 20	2	149	164			
-5 16	1	244	252	-4	2	2	315	334	-3	7	2	489	490	-1 12	2	120	-132	2 20	2	240	252			
-4 16	1	95	-79	-3	2	2	300	-294	-1	7	2	934	509	0 12	2	527	541	-1 21	2	326	310			
-3 16	1	267	263	-2	2	2	789	788	0	7	2	128	-124	1 12	2	205	187	-7 0	3	407	431			
-1 16	1	347	335	-1	2	2	106	-109	1	7	2	522	547	2 12	2	411	405	-5 0	3	552	548			
0 16	1	158	171	0	2	2	418	438	2	7	2	201	-206	4 12	2	225	217	-3 0	3	200	222			
1 16	1	320	323	1	2	2	227	-223	3	7	2	498	522	-5 13	2	344	352	-1 0	3	960	967			
3 16	1	266	262	2	2	2	509	509	5	7	2	295	315	-3 13	2	172	222	1 0	3	524	552			
-4 17	1	222	215	4	2	2	410	430	-6	8	2	332	332	-1 13	2	335	326	3 0	3	396	398			
-2 17	1	294	324	6	2	2	209	193	-4	8	2	406	475	1 13	2	737	737	5 0	3	281	267			
-1 17	1	181	184	-7	3	2	235	238	-2	8	2	716	715	3 13	2	138	113	-6 1	3	273	286			
0 17	1	355	364	-6	3	2	218	218	-1	8	2	163	165	5 13	2	241	224	-4 1	3	466	456			
2 17	1	279	264	-5	3	2	299	304	0	8	2	440	450	-4 14	2	265	242	-2 1	3	386	399			
3 17	1	74	50	-4	3	2	169	-170	2	8	2	625	644	-2 14	2	344	328	0 1	3	540	575			
4 17	1	265	280	-3	3	2	487	-488	3	8	2	79	-110	-1 14	2	79	-36	2 1	3	470	478			
-3 18	1	266	262	-1	3	2	672	661	4	8	2	506	500	0 14	2	512	498	4 1	3	284	280			
-1 18	1	253	245	0	3	2	363	374	5	8	2	107	68	2 14	2	253	234	6 1	3	220	209			
1 18	1	273	281	1	3	2	704	714	6	8	2	181	193	4 14	2	179	190	-7 2	3	253	266			
3 18	1	233	211	3	3	2	376	380	-7	9	2	275	281	-5 15	2	249	243	-5 2	3	390	396			
-2 19	1	391	391	5	3	2	336	323	-5	9	2	459	454	-3 15	2	209	221	-4 2	3	186	-183			
-1 19	1	127	-129	-6	4	2	276	271	-4	9	2	99	-105	-2 15	2	73	-52	-3 2	3	555	535			
0 19	1	150	139	-4	4	2	460	471	-3	9	2	516	532	-1 15	2	365	362	-2 2	3	455	442			
2 19	1	343	338	-2	4	2	941	935	-2	9	2	202	203	1 15	2	265	251	-1 2	3	627	615			
-3 20	1	302	307	-1	4	2	218	-217	-1	9	2	745	746	3 15	2	277	278	1 2	3	682	695			
-1 20	1	200	185	0	4	2	270	286	0	9	2	88	-60	-4 16	2	222	226	2 2	3	233	229			
1 20	1	231	220	2	4	2	685	664	1	9	2	435	449	-2 16	2	338	352	3 2	3	213	215			
-2 21	1	150	129	4	4	2	316	315	3	9	2	456	459	-1 16	2	80	85	5 2	3	283	280			
-1 21	1	86	-97	0	4	2	195	210	5	9	2	307	296	0 16	2	169	168	-6 3	3	296	305			
0 21	1	184	173	-7	5	2	335	347	-6	10	2	288	296	2 16	2	262	277	-4 3	3	581	572			
2 21	1	304	306	-5	5	2	179	160	-4	10	2	462	476	4 16	2	214	234	-2 3	3	536	542			
-6 0	2	189	202	-4	5	2	116	-115	-2	10	2	372	363	-4 17	2	98	-105	-1 3	3	223	212			
-4 0	2	500	505	-3	5	2	656	637	-1	10	2	195	-189	-3 17	2	234	236	0 3	3	846	845			
-2 0	2	259	271	-1	5	2	734	730	0	10	2	657	669	-2 17	2	134	146	1 3	3	114	93			
0 0	2	519	557	0	5	2	221	200	1	10	2	124	100	-1 17	2	215	223	2 3	3	369	373			
2 0	2	262	272	1	5	2	718	737	2	10	2	558	562	0 17	2	160	-154	4 3	3	305	310			
4 0	2	594	607	3	5	2	426	-407	4	10	2	337	336	1 17	2	250	250	5 3	3	126	-128			
6 0	2	162	179	3	5	2	456	448	6	10	2	232	242	3 17	2	218	209	6 3	3	263	255			
-7 1	2	135	135	5	5	2	317	321	-5	11	2	394	407	-2 18	2	276	274	-7 4	3	250	230			
-5 1	2	83	59	-6	6	2	349	325	-4	11	2	160	150	-1 18	2	116	-113	-6 4	3	161	170			
-5 1	2	593	587	-4	6	2	505	506	-3	11	2	383	387	0 18	2	182	167	-5 4	3	331	319			
-3 1	2	304	315	-2	6	2	687	687	-1	11	2	588	598	2 18	2	199	199	-3 4	3	409	405			
-2 1	2	84	76	-1	6	2	344	320	0	11	2	165	-147	3 18	2	123	114	-1 4	3	655	662			

OBSERVED AND CALCULATED STRUCTURE FACTORS

PAGE 3

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3	4	3	400	386	-3	10	3	516	532	2	16	3	123	-83	5	3	4	262	241
5	4	3	310	320	-1	10	3	474	494	3	16	3	226	221	-6	4	4	345	359
-6	5	3	268	266	0	10	3	88	-97	-4	17	3	174	169	-4	4	4	487	489
-5	5	3	296	297	1	10	3	474	461	-2	17	3	250	238	-2	4	4	489	499
-5	5	3	575	590	2	10	3	212	203	0	17	3	264	265	-1	4	4	155	147
-2	5	3	574	583	3	10	3	383	369	2	17	3	195	189	0	4	4	657	684
-1	5	3	171	157	4	10	3	157	-165	-3	18	3	243	229	1	4	4	76	-75
0	5	3	710	717	5	10	3	270	252	-1	18	3	260	284	2	4	4	399	397
2	5	3	532	530	-6	11	3	143	168	1	18	3	215	225	4	4	4	352	360
4	5	3	254	261	-4	11	3	473	480	-2	19	3	166	180	-7	5	4	215	204
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-7	6	3	298	289	-2	11	3	323	320	2	19	3	203	215	-3	5	4	593	577
-5	6	3	208	207	0	11	3	457	421	-1	20	3	191	217	-2	5	4	61	60
-4	6	3	118	116	2	11	3	471	463	1	20	3	185	191	-1	5	4	202	212
-3	6	3	534	547	3	11	3	267	-255	-6	0	4	331	341	1	5	4	427	429
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3	6	3	532	535	-5	12	3	247	-244	0	0	4	606	639	5	5	4	209	229
5	6	3	297	299	-3	12	3	441	435	2	0	4	584	585	-6	6	4	250	267
-6	7	3	401	416	-1	12	3	240	256	4	0	4	171	181	-4	6	4	400	375
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-4	7	3	259	259	3	12	3	252	262	-5	1	4	476	487	0	6	4	343	358
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-3	9	3	152	157	2	15	3	322	330	-6	3	4	139	-135	-1	9	4	482	492
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OBSERVED AND CALCULATED STRUCTURE FACTORS

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H	K	L	IOFC	IOFC	H	K	L	IOFC	IOFC	H	K	L	IOFC	IOFC
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-2	18	4	261	257	3	5	5	89	-83	-1	12	5	287	290
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5	0	5	326	332	-5	7	5	95	-104	-3	14	5	277	268
-6	1	5	226	213	-4	7	5	72	106	-3	14	5	200	206
-4	1	5	506	477	-3	7	5	89	-76	1	14	5	271	274
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-5	2	5	344	343	-5	8	5	219	223	-1	16	5	279	298
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-4	5	5	252	242	0	11	5	318	320	-2	2	6	203	206

OBSERVED AND CALCULATED STRUCTURE FACTORS

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M	K	L	IOFD	IOFC	M	K	L	IOFD	IOFC	M	K	L	IOFD	IOFC	M	K	L	IOFD	IOFC
-2	5	7	347	342	2	7	7	270	274	-4	11	7	259	261	1	1	8	233	233
0	5	7	264	265	-5	8	7	200	192	-2	11	7	250	263	-4	2	8	250	254
2	5	7	167	143	-3	8	7	239	236	0	11	7	220	219	-2	2	8	285	276
-5	6	7	265	256	-1	8	7	292	290	-3	12	7	255	251	-1	2	6	89	96
-3	6	7	226	242	1	8	7	244	240	-1	12	7	271	269	0	2	8	225	238
-1	6	7	405	418	-4	9	7	282	291	-2	13	7	149	157	-3	3	8	327	311
1	6	7	232	219	-2	9	7	300	307	-4	0	8	222	213	-1	3	8	182	194
2	6	7	130	111	0	9	7	290	295	-2	0	8	307	309	1	3	8	233	231
-4	7	7	288	316	-3	10	7	267	272	0	0	8	306	306	-4	4	8	235	217
-2	7	7	308	311	-1	10	7	250	264	-3	1	8	360	363	-2	4	8	180	199
0	7	7	251	241	1	10	7	197	205	-1	1	8	145	154					

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